

***AC SUSCEPTIBILITY SETUP
FOR 7K CLOSE CYCLE REFRIGERATOR***

**A THESIS SUBMITTED IN PARTIAL FULFILMENT OF THE
REQUIREMENTS FOR THE DEGREE OF**

Master of Science in Physics

By

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Under the supervision of

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CERTIFICATE

This is to certify that the dissertation entitled, “***AC SUSCEPTIBILITY SETUP FOR 7K CLOSE CYCLE REFRIGERATOR***” submitted by Miss Tripta Parida for the award of Master of Science in Physics (2010-12) in the Department of Physics, National Institute of Technology, Rourkela, is a record of authentic work carried out by her under my supervision. To the best of my knowledge, the results embodied in this dissertation have not been previously submitted for any degree in this/any other institute.

Date:

Dr. Prakash Nath Vishwakarma

DECLARATION

I, Tripta Parida do hereby declare that, this dissertation titled “*AC SUSCEPTIBILITY SETUP FOR 7K CLOSE CYCLE REFRIGERATOR*” is an authentic work done by me in the Department of Physics, NIT Rourkela. I also declare that this report has been neither published before nor submitted to any other institution.

Tripta Parida

Abstract

Cryocooler based ac susceptometer is designed. Ac susceptibility is measured using the mutual inductance principle. The setup made of hylum consists of primary coil, secondary coil and a sample holder. Copper wire (150micron) winding is done on the primary and the secondary coils. The secondary coil is connected in series opposition i.e. if one wound is clockwise, the other is anticlockwise. Lock-in amplifier excite the primary coil, and the signal from secondary is given to the lock-in-amplifier itself where the in-phase and out-phase components are separated. Lakeshore Temperature controller is used for the detection of cryocooler temperature and a Pt100 temperature sensor is used for the detection of sample temperature. This is because as the distance between the cryocooler and the sample is more, so cryocooler temperature sensor is not able to detect the sample temperature. The temperature controller and lock-in-amplifier is then interfaced with computer using Lab-View program. Then, LSMO sample is prepared for testing the working of set up. Sample is prepared by auto combustion sol-gel method. Pellets are made which is cut into rectangular shape and placed in the centre of one of the secondary coil. Then the ac susceptibility measurement is done by keeping the frequency constant (834 Hz) in lock-in amplifier. Data are plotted and the susceptibility plot with sample is obtained. The susceptibility plot for sample was obtained by deducting the background from the susceptibility plot with sample. The phase change in the LSMO system is studied using the Ac susceptometer.

CHAPTER 1

INTRODUCTION

Scientists believe it was the Greeks who first reflected upon the wondrous properties of magnetite, the magnetic iron ore $\text{FeO-Fe}_2\text{O}_3$ and named lodestone. From 800 B.C. magnets were in use, and magnetism was experienced in day to day life [1]. Magnetism is a property of materials that respond to an applied magnetic field. All materials are influenced varyingly by the presence of a magnetic field. Some are attracted to a magnetic field (paramagnetism and ferromagnetism); others are repulsed by a magnetic field (diamagnetism); others have a much more complex relationship with an applied magnetic field (spin glass behaviour and antiferromagnetism, ferrimagnetism). Substances that are negligibly affected by magnetic fields are known as **non-magnetic** substances.

Though it seems magnetism is prevalent in classical world but its origin is quantum mechanical in nature. The magnetism in solids arises due to orbital and spin motions of electrons as well as spins of the nuclei. This gives the concept of magnetic moment. The magnetic moment associated with orbital and spin can be defined as Orbital angular magnetic moment(L) and spin Magnetic moment (S) respectively[2].The total magnetic moment is sum of orbital and spin magnetic moment. The Magnetization of the material can be defined as total magnetic moment (M) per unit volume defined by equation.1. 1

$$\mathbf{M}=(N/V)\mathbf{m}= n\mathbf{m} \dots\dots\dots 1.1$$

The quantity N/V is usually written as n , the number density of magnetic moment. Net magnetization results from the response of a material to an external magnetic field.

Sometimes, the magnetisation measurements lack information's (i.e. the dynamics of magnetic systems), so magnetic susceptibility measurement is a good hand practice to obtain qualitative information about the material.

The magnetic susceptibility χ_m is a dimensionless proportionality constant that indicates the degree of magnetization of a material in response to an applied magnetic field,

The magnetic susceptibility of most crystals is not a scalar. Magnetic response \mathbf{M} is dependent upon the orientation of the sample and can occur in directions other than that of the applied field \mathbf{H} . In these cases, volume susceptibility is defined as a tensor

$$\mathbf{M}_i = \chi_{ij} \mathbf{H}_j \dots\dots\dots 1.2$$

where i and j refer to the directions (e.g., x and y in Cartesian coordinates) of the applied field and magnetization, respectively. The magnetic susceptibility is thus a tensor of rank 2, describing the component of magnetization in the i -th direction from the external field applied in the j -th direction[1].

From the basic study of magnetism, the magnetic permeability and magnetic susceptibility are related by equation 1.3

$$\mu_r = 1 + \chi_m \dots\dots\dots 1.3$$

1.1 MAGNETIC BEHAVIOUR OF MATERIALS

Depending on this susceptibility, there are basically three kinds of magnetic materials (diamagnetic, paramagnet, ferromagnetic). Other two (antiferromagnetic, ferrimagnet) are related to spin alignment. The details of it are given below:-

Diamagnetism:

Diamagnetic materials have a very weak *negative* susceptibility, typically of order -10^{-6} . That is to say, the relative permeability is slightly *less than* 1. The magnetization verses Magnetic field and susceptibility verses temperature is described in fig.1. The figure 1(a) shows a negative susceptibility for diamagnetic material and 1(b) indicates it is temperature independent. The examples of some materials exhibiting diamagnetism are, Bi, Hg, Ag, Pb, Cu, H₂O, etc.[2]

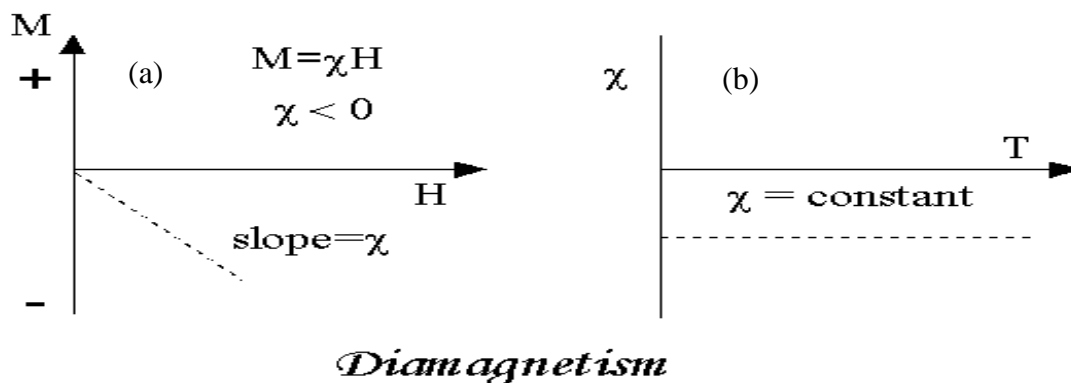


Figure.1.1 shows a) M vs. H and b) χ vs. temperature plots for diamagnetic materials [3]

Paramagnetism:

Some atoms or molecules have a permanent magnetic moment. The magnetic moment of an atom of a molecule is typically of order of $+10^{-5}$. The presence of a permanent magnetic moment is often the result of *unpaired electron spins*. When a paramagnetic material is placed in a magnetic field, the magnetic moments experience a torque and they tend to orient themselves in the direction of the magnetic field, thus augmenting, rather than diminishing, B . In figure 1.2 (a) M verses H plot shows that Magnetisation is directly proportional applied

field and 1.2(b) shows that the paramagnetic susceptibility is a function of temperature and decreases with increase in temperature (Curie's law). The examples of some paramagnetic materials are Na, gaseous nitric oxide, O_2 , Mn^{+2} , etc.[2]

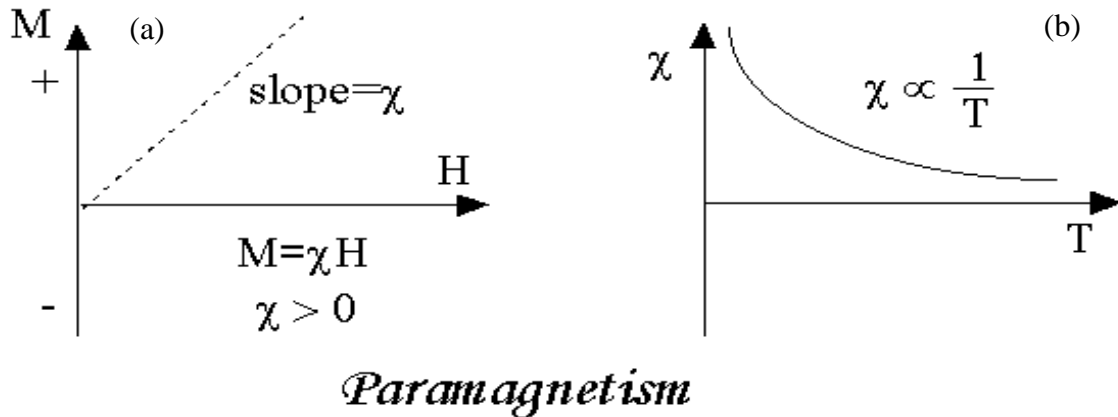


Figure.1.2 shows a) M vs. H and b) χ vs. temperature plots for paramagnetic materials [3]

Ferromagnetism:

What we normally think of as magnetic materials are technically *ferromagnetic*. The susceptibilities of ferromagnetic materials are typically of order $+10^3$ or 10^4 or even greater. However, the ferromagnetic susceptibility of a material is quite temperature sensitive, and, above a temperature known as the *Curie temperature*, the material ceases to become ferromagnetic, and it becomes merely paramagnetic. Within a single crystal, there exist *domains*, within which all the magnetic moments are parallel and are aligned with a particular axis. In an adjacent domain, again all the moments are parallel to each other, but they may be aligned with a *different* axis. Thus we have a number of domains, each highly magnetized, but with some domains magnetized in one direction and some in another. The domains are separated by domain boundaries, or “Bloch walls”. Figure 1.3 is a schematic sketch of a crystal divided into domains, with the magnetization in a different direction in each and with varying field alignment of the domains. The examples of ferromagnetic materials are the elements such as Fe, Co, Ni, Gd, and a number of alloys and oxides such as MnBi, MnAs, CrO_2 , etc. [2]

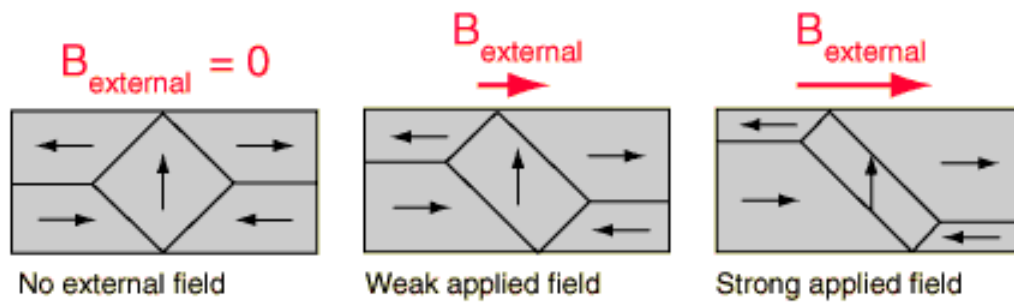


Figure.1.3 shows a schematic sketch of a crystal divided into domains for ferromagnetic materials and the domain alignment with varying field[1].

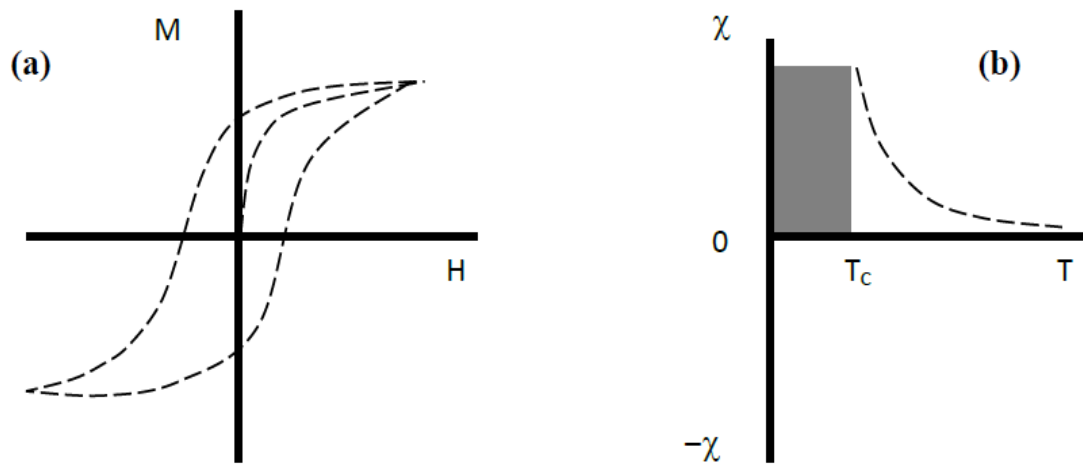


Figure.1.4 shows (a) the M-H loop and (b) χ vs. temperature behavior of ferromagnetic materials. Below curie temperature (T_c) the material is ferromagnetic and above T_c the material behaves as paramagnet[3].

Antiferromagnetism:

Antiferromagnetic substances have a small susceptibility at all temperatures, but their susceptibility vary in a peculiar way. As shown in figure 1.5, with decrease in temperature, χ increases but finally goes through a maximum at a critical temperature T_N called Neel Temperature. The substance is paramagnetic above T_N and antiferromagnetic below it. The examples of antiferromagnetic materials are the MnO , FeO , NiO , FeS , FeCl_2 etc.[3]

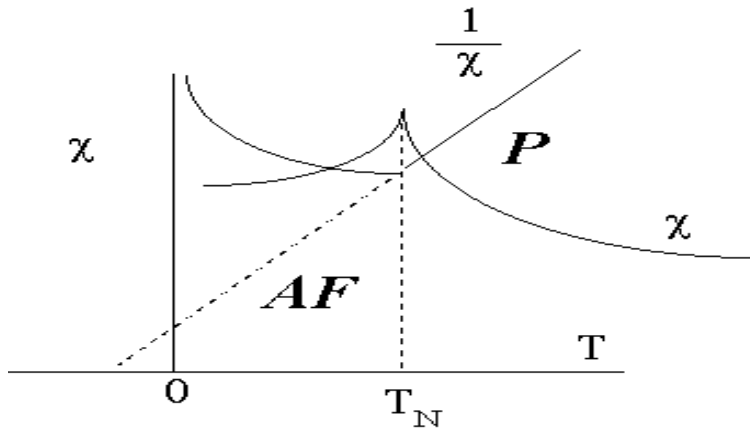


Figure.1.5 Variation with temperature of the susceptibility χ , and of $1/\chi$ for an antiferromagnetic. AF= antiferromagnetic, P= Paramagnetic [3].

Ferrimagnetism:

It is similar to antiferromagnetism but except that adjacent moments are unequal in magnitude and hence complete cancellation of moment does not takes place. Again like ferromagnetic, they exhibit the phenomenon of hysteresis as shown in fig 1.6(a). Their spontaneous magnetisation disappears above a certain temperature T_N , called Neel temperature, and they become paramagnetic as shown in fig 1.6(b)

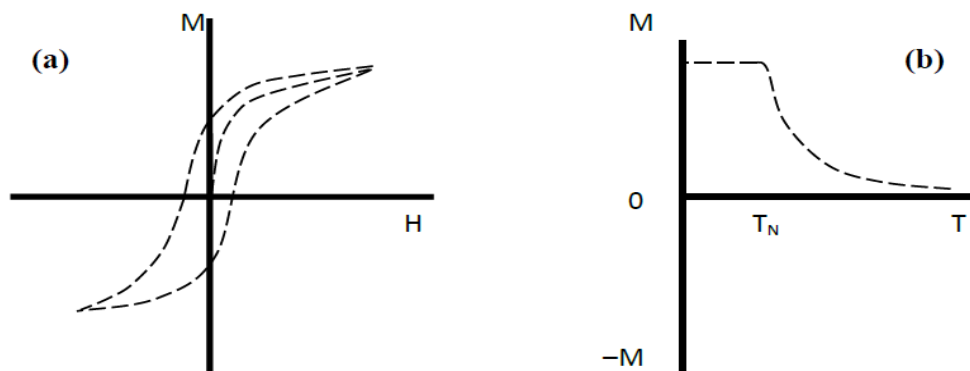


Figure.1.6 shows the (a) M-H loop and (b) M-T response of ferrimagnetic material. Below Neel temperature (T_N) the material is ferrimagnetic and above T_N it is paramagnetic [3]

1.2 MEASUREMENT OF AC SUSCEPTIBILITY:

There are mainly two methods used to extract the susceptibility of any material, i.e., ac and dc method. In general, the sample is exposed to a magnetic field (constant or time varying) and sample magnetization is measured. The magnetization is usually measured by flux extraction technique which is based on Faraday's law. It is based on the flux change in magnetic lines of force through coil of area A. In this method, the change in flux $d\phi$ is measured as induced voltage $v(t)$ in the secondary coil.

$$v(t) = \frac{d\phi}{dt} \dots\dots\dots 1.4$$

Since the flux is $\phi = BA$, where B is the magnetic induction and A is area of loop.

Hence, the expression for induced voltage becomes:

$$v(t) \propto \frac{d(BA)}{dt} \Rightarrow v(t) \propto A \frac{dB}{dt} + B \frac{dA}{dt} \dots\dots\dots 1.5$$

The first term, where the area of the loop A is constant and the magnetic induction varies with time is called the AC method. The second term where the magnetic induction B remains constant whereas the change in flux is due to change in the area of the loop, is called DC method. In practice, the area is not changed directly, but indirectly via vibrating the sample.

DC method

In the **dc method**, the measured parameter is magnetization, "M" which may be converted to susceptibility " χ " using the relation,

$$\chi_{dc} = M/H$$

where "H" is the applied magnetic field. The dc-magnetometer and the ac susceptometer are two entirely different tools that provide different ways of examining magnetic properties. Both these two techniques rely on sensing coils used to measure the variation in the magnetic flux due to magnetic sample.

In a dc magnetization measurement a value for the magnetization "M" is measured for some applied dc field, H_{dc} [4]. If the sample being measured does not have a permanent magnetic moment, an applied field is required to magnetize the sample. A detection coil is used to detect the change in magnetic flux due to the change in magnetic moment of the sample.

Since the applied magnetic field is constant, there will be no signal associated with H_{dc} (Faraday's law). The dc or static susceptibility is thus given by:

$$\chi_{dc} = M / H_{dc}$$

A variation of this method involves 2 identical search coils (coil a and b), located symmetrically in the solenoid. These are connected in “series opposition” i.e., if one is wound clockwise, viewed along their common axis, the other is wound anticlockwise. If area –turns of both coils are equal, change in H will induce equal and opposite emf's in 2 coils, and the fluxmeter will show no deflection. Thus variations in the applied field will not influence the results. Initially the sample in coil a, and the magnetic flux which the coil a measures the magnetic flux $B_s A = \mu (H+M)A$. When the specimen is moved out of coil a, then the coil a measures the magnetic flux as $B_s A = \mu_0 H A$. Then the sample enters into coil b, and the measured flux by b is $B_s A = \mu_0 (H+M)A$.

Because of the presence of second coil (b), the signal is twice that obtained with a single coil.

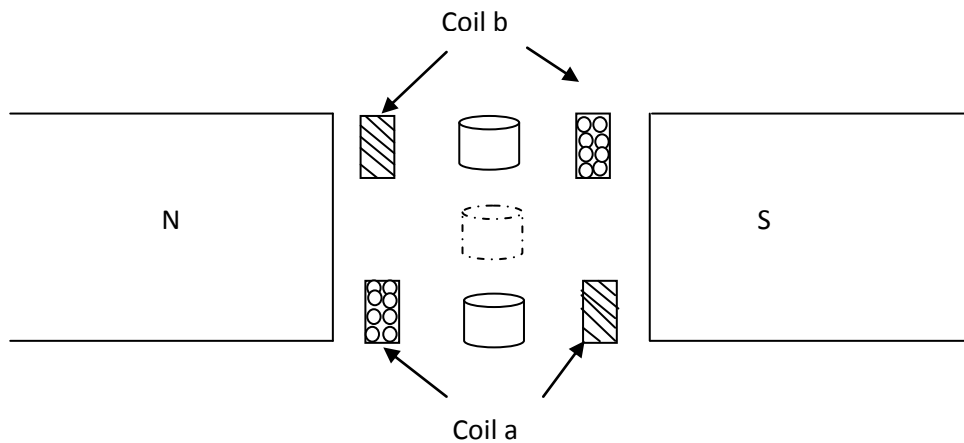


Figure 1.7: Schematic of dc susceptibility measurement

ac method:

Though the principle behind dc magnetometer and ac susceptometer is detection of magnetic flux, the main difference lies in how the flux variation is achieved. In ac susceptometer the sample is magnetized by ac magnetic field H_{ac} . The flux produced by the sample is sensed by the detection coil. The magnetic moment of the sample generally follows the applied field. The detection circuitry is generally balanced with, a second identical but oppositely wound coil, to null out the flux changes related to H_{ac} . As a result, any experimentally detected change in flux is only due to the changing magnetic moment of the sample as it responds to the ac field (no sample movement is required to produce an output signal) and not to the moment itself as in dc technique. The ac susceptibility is:

$$\chi_{ac} = dm / VH_{ac} \rightarrow dM/dH$$

Thus, the ac susceptibility is actually the slope (dM/dH) of the magnetization curve (M versus H curve). The ac technique detects changes in the magnetization that lead to dM/dH in the limit of small ac fields, and this is why sometimes referred to as a differential susceptibility. This is the fundamental difference between the ac and dc measurement techniques.

Ac susceptibility is most conveniently measured using the mutual inductance principle. A sample is subject to a small alternating field, $H(t) = H_{ac} \sin \omega t$ produced by a primary coil, and the resulting emf induced in a secondary coil wound around the sample is detected. The emf $v(t)$ is directly proportional to the time derivative of the magnetisation of the sample. The time dependent volume magnetisation $MV(t)$ can be expressed as a Fourier series of non-linear complex ac susceptibility $\chi' + i\chi''$ as:

$$M_V = H_{ac} \sum_n \chi'_n \sin n\omega t + \chi''_n \cos n\omega t \dots \dots \dots 1.6$$

In almost all situations, two identical secondary coils are positioned symmetrically with respect to the primary coil and are connected in series opposition such that the voltage induced either directly by the ac field H_{ac} or indirectly by background sources H_{bg} are effectively and precisely nulled. With this arrangement the total time dependent induced voltage across the two identical secondary coils is

$$V(t) = -\frac{d\phi}{dt}$$

Where the total flux is given by,

$$\Phi(t)=[B_a-B_b]A = \pi r_{ab}^2 n_{ab} [\mu_0 \{M_V(t)+H_{ac}(t)+H_{bg}(t)\}_a - \mu_0 \{H_{ac}(t)+H_{bg}(t)\}_b]$$

$$\Rightarrow \Phi(t) = \mu_0 \pi r_{ab}^2 n_{ab} (M_V(t))_a$$

If and only if $(H_{ac}(t)+H_{bg}(t))_a = (H_{ac}(t)+H_{bg}(t))_b$. $V/L = \pi r_{ab}^2$, r_{ab} and n_{ab} are the radii and number of turns of the 2 secondary coils (a & b). The sample is positioned in coil a. Hence the induced voltage takes the form

$$V(t) = -\frac{d\phi}{dt} = -\mu_0 V N H_{ac} \frac{d}{dt} [\sum_n \chi_n' \sin n\omega t + \sum_n \chi_n'' \cos n\omega t] \dots \dots \dots 1.7$$

$$V(t) = \mu_0 V N n \omega H_{ac} \sum_{n=1}^{\infty} [\chi_n'' \sin n\omega t - \chi_n' \cos n\omega t] \dots \dots \dots 1.8$$

Where, it is assumed that the sample of volume V fills the whole of the secondary coil a and that $N=n_s/L$ where L is the length of a and b.

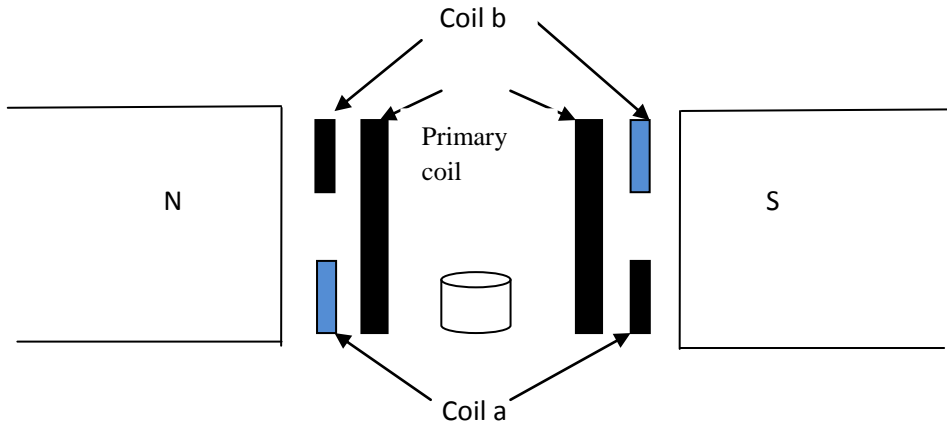


Figure 1.8: Schematic of ac susceptibility measurement

It is clear from the equation that the emf $V(t)$, induced in the secondary coil.

The real and imaginary component of susceptibility χ_n' and χ_n'' are determined directly from $M(t)$ through the relationship

$$\chi_n' = \frac{1}{\pi H} \int_0^{2\pi} M(t) \sin(n\omega t) d(\omega t) \dots \dots \dots 1.9$$

$$\chi_n'' = \frac{1}{\pi H} \int_0^{2\pi} M(t) \cos(n\omega t) d(\omega t) \dots \dots \dots 1.10$$

N=1 denotes the fundamental susceptibility while n= 2, 3, 4... etc are the higher order harmonics associated with non-linear terms in χ .

DIFFERENCE BETWEEN AC AND DC MEASUREMENT:-

In the dc measurement, the magnetic moment of the sample does not change with time. Thus, a static magnetic measurement is performed. In the ac measurement, the moment of the sample is actually changing in response to an applied ac field, allowing the dynamics of the magnetic system to be studied [4].

1.3 WHY AC SUSCEPTIBILITY?

It has a great application in different fields, like spin glass, superparamagnetism, superconductivity etc. A brief discussion is given below.

(I)SPIN-GLASS

Spin glass can be defined as random, interacting, magnetic systems characterized by random, yet co-operative, freezing of spins at a well- defined temperature T_f below which a highly irreversible, metastable frozen state occurs without the usual long-range spatial magnetic ordering [5]. ‘Co-operative’ implies ‘ordering’ or even a phase transition. Spin glass represent the class of material exhibiting the frozen-state transition. Usually this behaviour is characterized by ac susceptibility [6]. Below T_f the material exhibits spin glass behaviour and above it is paramagnetic. The freezing temperature is determined by measuring χ' vs. temperature, and is basically represented by a cusp at the freezing temperature. The location of the cusp is dependent on the frequency of the AC susceptibility measurement, a feature that is not present in other magnetic systems and therefore confirms the spin-glass phase.

II) SUPERPARAMAGNETISM

Particles whose magnetization changes spontaneously are analogous to paramagnetic atoms, but if their magnetic moment is much larger, then it refers to superparamagnetism[7]. Here, the particles exhibit single-domain ferromagnetic behavior. Below the blocking temperature T_B , the material exhibits superparamagnetism and above it is paramagnetism. AC susceptibility measurement is an important tool for determining this blocking temperature T_B .

(III) SUPERCONDUCTIVITY

Resistivity versus temperature plot can give the transition temperature from normal to a superconducting behavior, but whether it is a surface or bulk phenomenon that can only be determined by ac susceptibility measurement.

In the normal state (above the critical temperature), superconductors typically have a small susceptibility. In the fully superconducting state, the sample is a perfect diamagnet and so $\chi' = -1$. Typically, the onset of a significant nonzero χ' is taken as the superconducting transition temperature, which can be measured by ac susceptibility measurement.

The various applications of ac susceptibility have installed a motivation to design and fabricate an ac susceptometer. For this purpose we have reviewed many literatures, of which one is discussed.

CHAPTER-2 DESIGN AND FABRICATION OF SETUP

2.1 DESIGN OF A TYPICAL SUSCEPTOMETER:

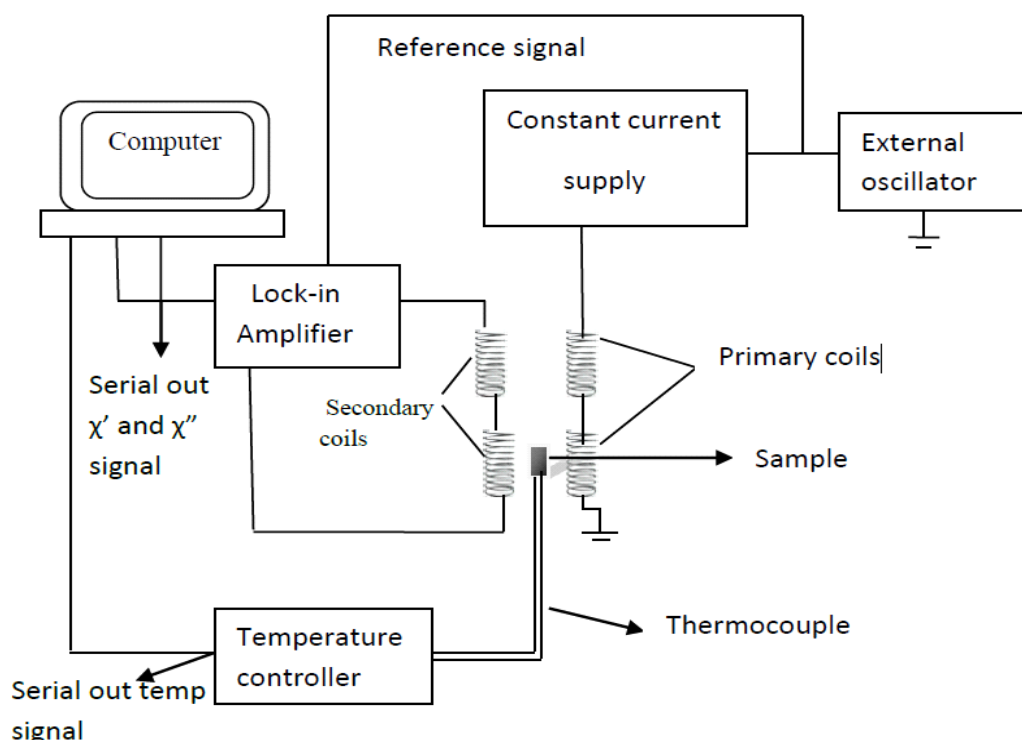


Fig 2.1 shows the block diagram of an ac susceptometer [8]

In the above figure, the schematic diagram of an ac susceptometer designed by [8] is shown. It is a four coil system which consists of two identical primary excitation field coil of 120 turns each and two identical secondary pick-up coils of 600 turns each. The coils are wound around a cylinder of 1.2 cm in height [8]. The current is passed by a constant power supply which is coupled to an external oscillator and is sent through the primary coil. This current generates a magnetic field which magnetizes the sample and the flux change in sample is detected by the secondary coil. The signal from this secondary coil goes to a computer controlled lock in amplifier where the in-phase and out of phase components of complex ac susceptibility are separated.

Former Material selection [8]:

The chosen materials should be:

1. Non magnetic
2. Poor electrical conductors
3. Good thermal conductors
4. Minimal thermal expansion properties

2.2 CROSS-SECTIONAL VIEW OF TWO DESIGNS:-

Motivation for design no.1:

Earlier the experiment for ac susceptibility was performed for high temperature measurements. So, now it was a good hand practise to measure the same at low temperature. For a trial basis, a similar design was made and experiment was performed. The figure given below shows the cross-sectional view of design 1. Figure 2.2 is for the secondary and 2.3 is for primary coil.

DESIGN NO.1

Secondary coil:

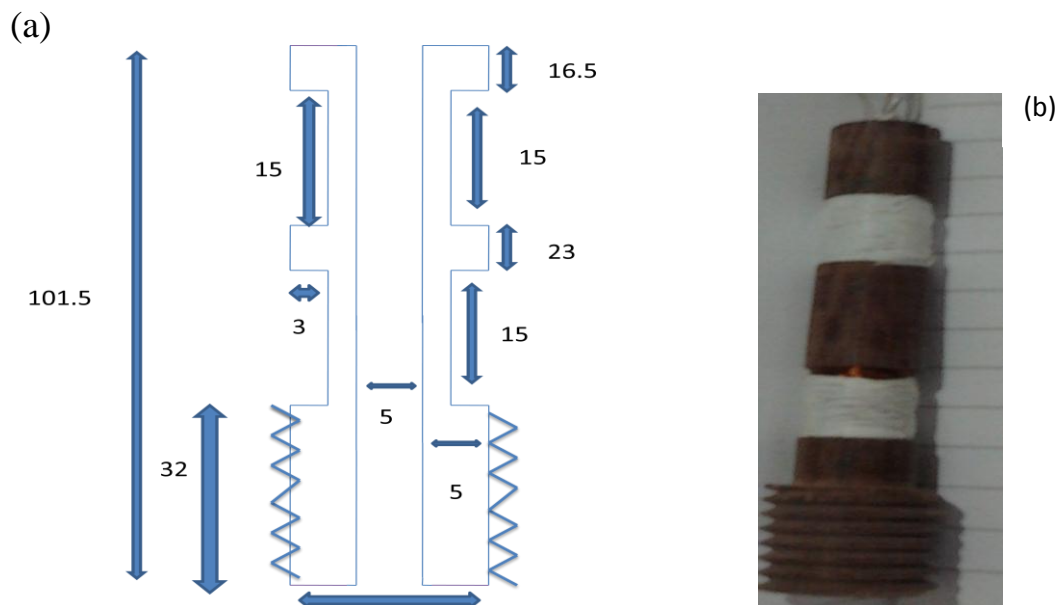


Fig 2.2 (a) shows the cross sectional view of the secondary coil of design 1.and (b) shows the schematic view. All parameters are in mm.

Primary coil:

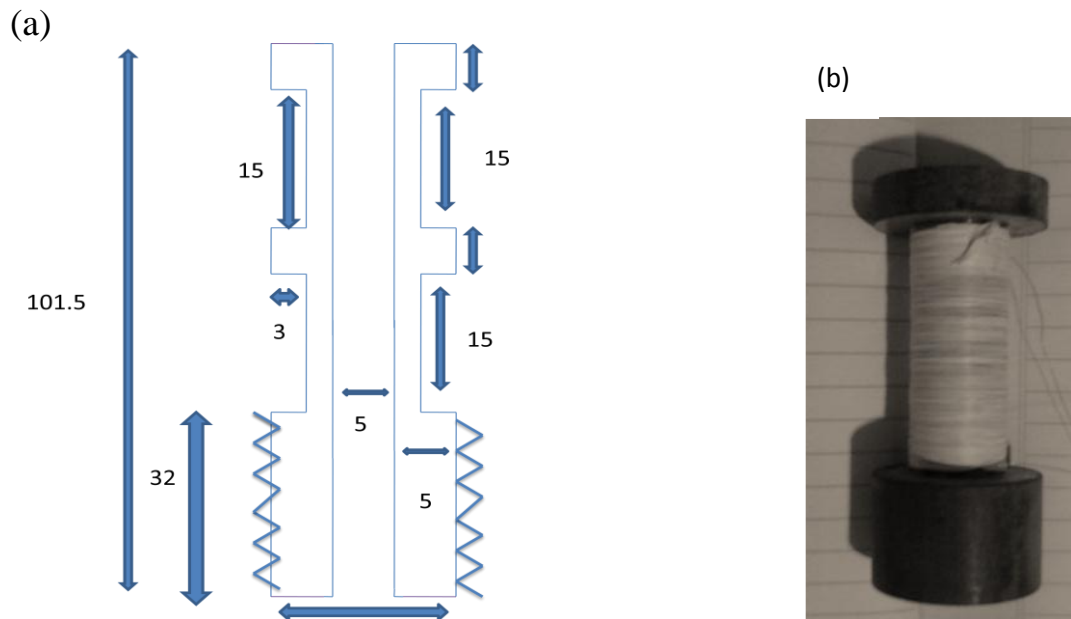


Fig 2.3 (a) shows the cross sectional view of the primary coil of design 1. and (b) shows the schematic view. All parameters are in mm

The material chosen for all this is hylum because of its high thermal conductivity and low electrical conductivity which can avoid eddy current. After the design, copper winding (150 micron) is done over primary and secondary coils. 1200 turns of copper wire is wound over primary. The secondary coil is a two coil system and the coiling area is separated by some distance 5mm. It had 500 turns wound in clockwise direction and another 500 turns in counter-clockwise direction. The secondary coil was inserted into the primary coil and the sample holder was inserted into the secondary pick up coil. A Pt100 temperature sensor is placed inside the sample holder, in contact with the sample, for measuring the sample temperature. This set-up was dipped in liquid nitrogen and with the help of lock-in-amplifier voltage was measured, which was then converted to susceptibility using relation 1.9 and 1.10.

Motivations for design 2:

With the help of 1st design we were able to measure the ac susceptibility between the temperature range from room temperature (300 K) to liquid nitrogen temperature (77 K). The installation of the Closed Cycle Refrigerator (CCR) in our lab has motivated us to design a cryocooler based ac susceptometer. The advantage is that we can lower the temperature down to liquid helium temperature (4 K). For second design we have reduced the dimensions of

primary and secondary coil system so that it could be fit in cryocooler. The numbers of turns in primary and secondary coils are adjusted according to it. The number of turns in the primary coil is 3000 and in secondary the number of turns is 1800 clockwise and 1800 anticlockwise. The secondary is inserted into the primary and a pt-100 sensor is used for the detection of sample temperature. The cross-sectional view of 2nd design is shown in fig 2.4.

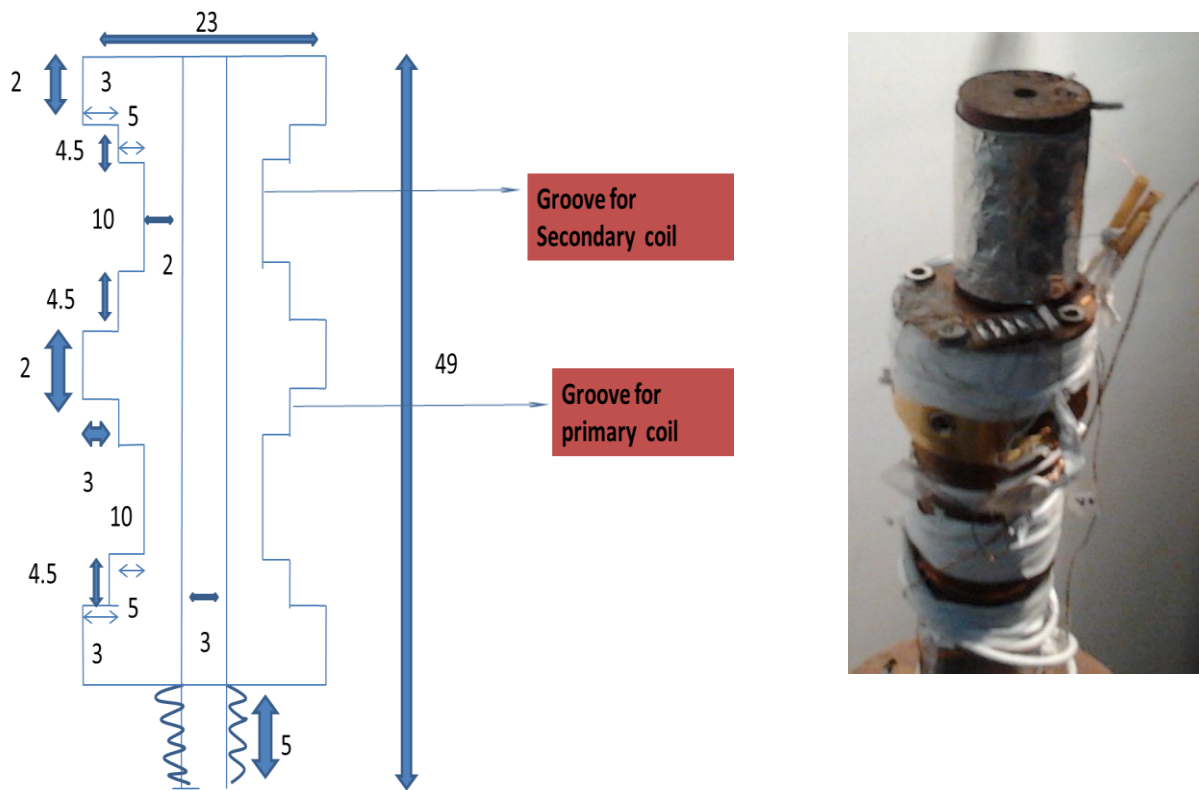


Fig 2.4 (a) shows the cross sectional view of the primary coil and secondary coil of design 2. and (b) shows the schematic view. All parameters are in mm

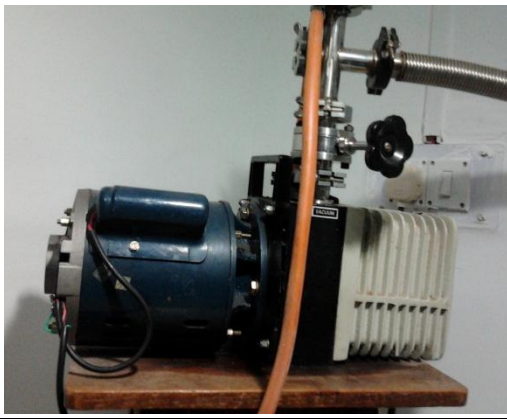
Instruments used for measurements :-

1. Lock-in- amplifier phasing:



The lock-in-amplifier acts as a discriminating voltmeter. It measures the amplitude and the relative phase of an ac signal. one of the most useful features of the ac susceptibility is that both the real or in-phase component χ' , and the imaginary or out of phase component χ'' can be measured.

2. Vacuum pump :



Vacuum helps for the insulation.

3. Compressor:



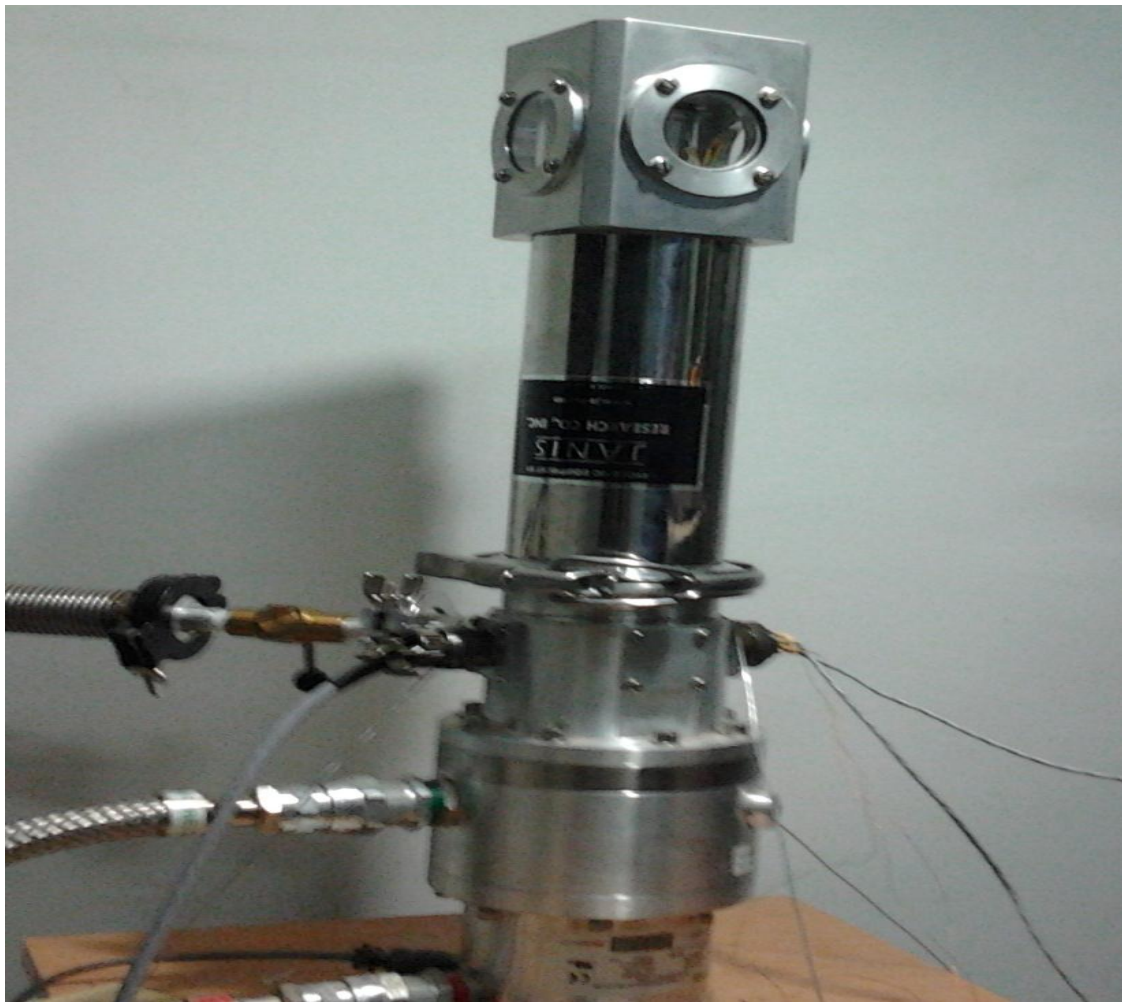
Warm gas is introduced into the compressor through the inlet port, where it releases heat which is absorbed by the helium gas present inside the compressor. The resulting cooled gas then exits the compressor through the outlet port.

4 . Temperature controller:



Using this temperature controller we could determine the temperature of cryo cooler.

5. Cryocooler



2.3 Calculation for the conversion of voltage to susceptibility:-

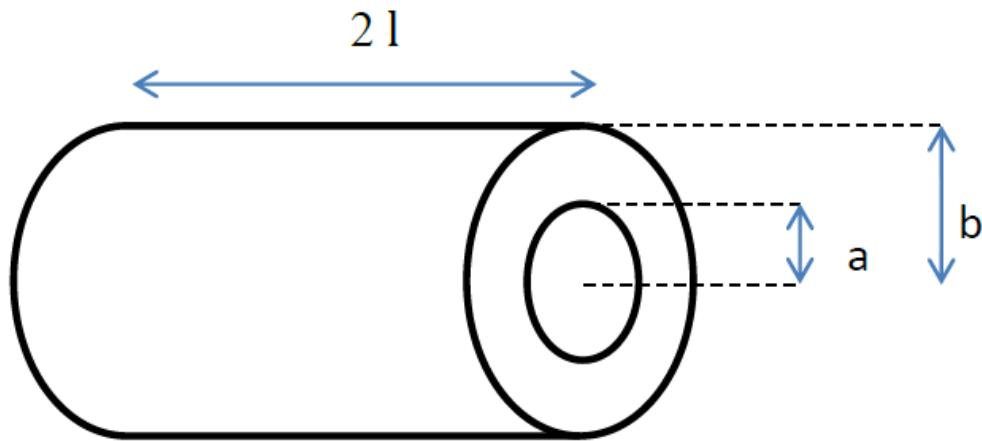


Fig 2.5 Dimensions of solenoid

The magnetic field at the centre of the solenoid is given by :-

$$H_{ac} = J a F(\alpha, \beta) \dots\dots\dots 2.1$$

where $F(\alpha, \beta)$ is the field factor which depends on the cross-sectional shape of solenoid.

$$F(\alpha, \beta) = \beta \ln \frac{\alpha + \sqrt{\alpha^2 + \beta^2}}{\sqrt{1 + \beta^2}} \dots\dots\dots 2.2$$

Where

$$\alpha = b/a$$

and

$$\beta = l/a$$

where a and b are the inner and outer radius of solenoid respectively and $2l$ is the length of the solenoid.

For the set-up we have designed,

The value of :

$$a = 8.5, \quad b = 11.5mm \quad \text{and} \quad l = 22mm.$$

Putting these values we get, $\alpha = 1.353$

$$\text{and} \quad \beta = 2.588$$

So, the value of $F(\alpha, \beta)$ comes to

$$F(\alpha, \beta) = 0.3186$$

The calculated amount of current passing through the solenoid is $I = 5\mu\text{A}$.

So, the average current density

$$J = I/A$$

$$A = 2(b-a)$$

$$= 132 * 10^{-6} \text{ m}^2$$

The value of J comes to $J = 3787 \text{ A/m}^2$.

$$B = \mu_0 H_{ac} = 1.289 * 10^{-7} \text{ Tesla}$$

The measured rms voltage V across the two secondary coil is :-

$$V(t) = -d\phi/dt$$

$$V_0 = \mu_0 \pi a^2 \omega N H_{ac} \dots\dots\dots 2.3$$

Hence, $V_0 = 4.72 * 10^{-4} \text{ volts.}$

$$\chi'_n = \frac{v(x)}{v_0} \dots\dots\dots 2.4$$

$$\chi''_n = \frac{v(y)}{v_0} \dots\dots\dots 2.5$$

CHAPTER-3

Sample Preparation

3.1 Sample Selection

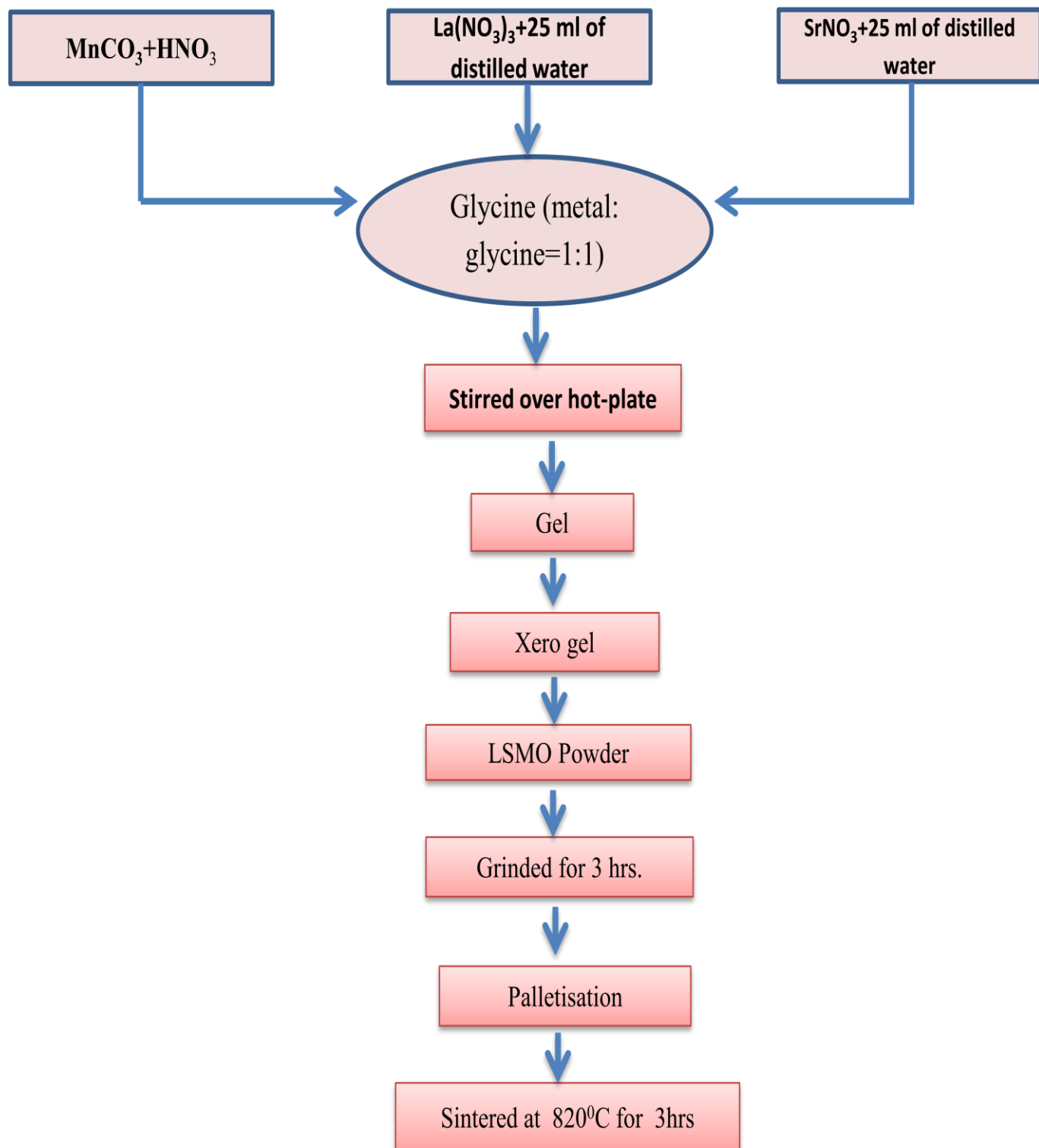
We have chosen $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$ as sample as its transition temperature is within the limits of 100K~300K . $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$ has immense applications in different fields of physics. It is the derivative of parent compound LaMnO_3 . Sr doping in antiferromagnetic insulator LaMnO_3 at La site enhances ferromagnetic interactions in the compound LaMnO_3 . [9]

3.2 LSMO Preparation:

LSMO powders are prepared by auto combustion sol-gel method. $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, $\text{Sr}(\text{NO}_3)_2$ and MnCO_3 are chosen as precursors for the synthesis. Stoichiometric amounts are taken as 0.83% of 0.01mole, 0.17% of 0.01mole, 0.01mole respectively. Combustion agent used in synthesis is Glycine. The ratio of glycine: metal nitrate is 1:1.

The reactants are dissolved in distilled water and continuously stirred till a colourless solution is obtained. Then glycine is added to the solution and is then heated with constant stirring at 80°C to evaporate the excess solvent. The viscous solution after 3 hour of heating became a viscous gel. The gel converted to the black powder after burning due to glycine agent. The powder is collected and grinded for 3 hours. Then it was calcined at 600°C for 2 hours. The furnace heating rate is maintained at $4^\circ\text{C}/\text{minute}$. After cooling the sample was collected and grinded for another 3 hours. Pellets are made from the sample and sintered at 820°C for 3 hours. After cooling the pellets are collected. Pellets are cut into small rectangular shaped pieces and wrapped in Teflon tape for use in ac susceptometer in low temperature. Finally susceptibility measurement is done using ac susceptometer already fabricated.

Flow chart to prepare LSMO:



Chapter 4

RESULT AND DISCUSSION

4.1 AC SUSCEPTIBILITY OF LSMO:

The sintered pellets are cut into rectangular pieces and are wrapped with Teflon tape. One of these rectangular pieces is then carefully placed at the centre of one of the secondary coil in order to avoid the edge effect. The set up is cooled down in cryocooler. The set-up is cooled down to 10 K and then allowed to heat up normally to room temperature. The x- component (in phase) and y- component (out of phase) of signal are then recorded by lock in amplifier as a function of temperature. These x and y component is then converted to volume susceptibility by dividing with parameter v_0 (eq 2.4 and 2.5). Finally the in-phase and out-of-phase components of complex ac susceptibility are plotted against temperature. The variation of in-phase (χ') and out-of-phase (χ'') susceptibilities with temperature is shown in Fig 4.1 and Fig 4.2 respectively. Then the background data is subtracted from the susceptibility data with sample to get the in phase and out of phase components of ac susceptibility due to the $\text{La}_{0.83}\text{Sr}_{0.17}\text{MnO}_3$ sample. Results are shown in Fig 4.3 and Fig 4.4.

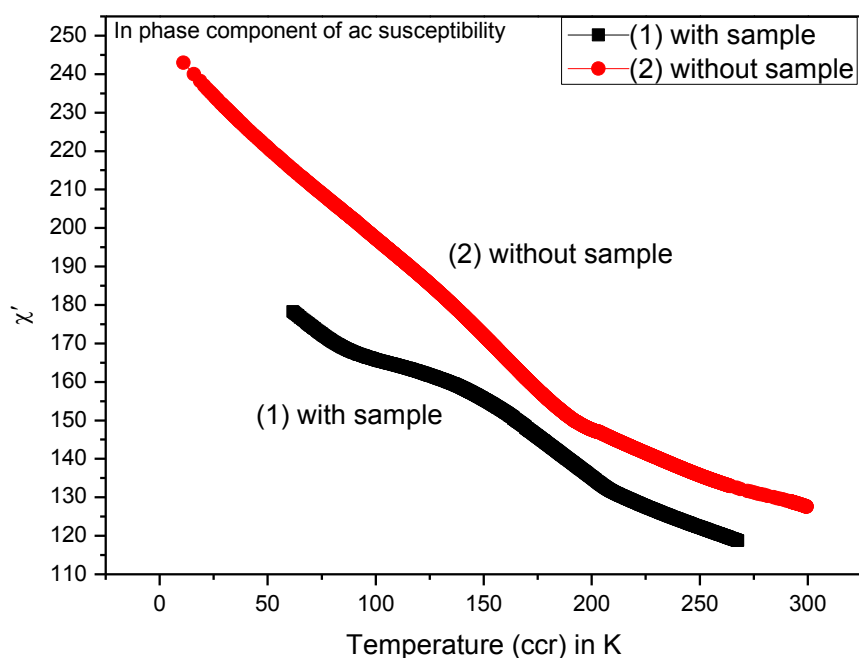


Fig 4.1 Shows the In-phase component of ac susceptibility for both with sample and without sample.

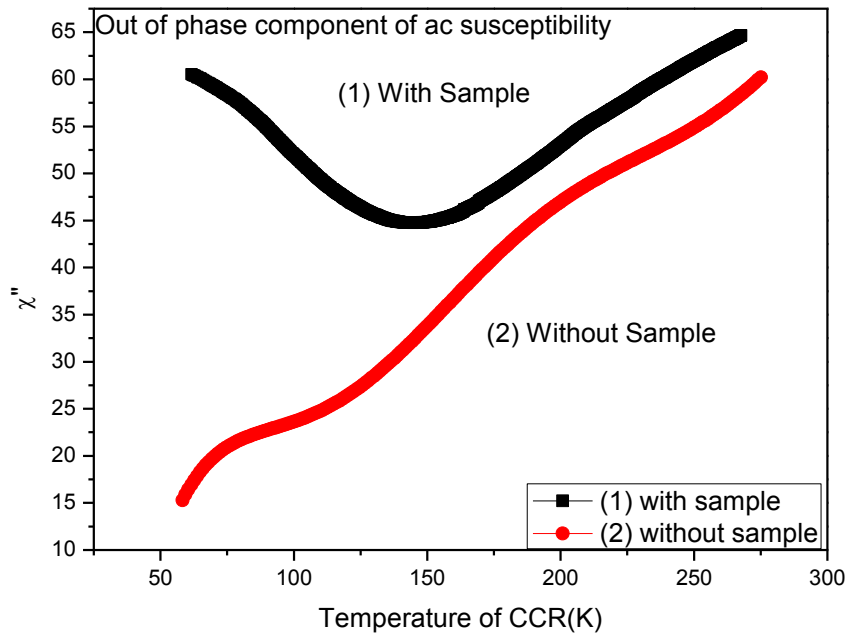


Fig 4.2 shows the out-of-phase component of ac susceptibility for both with sample and without sample.

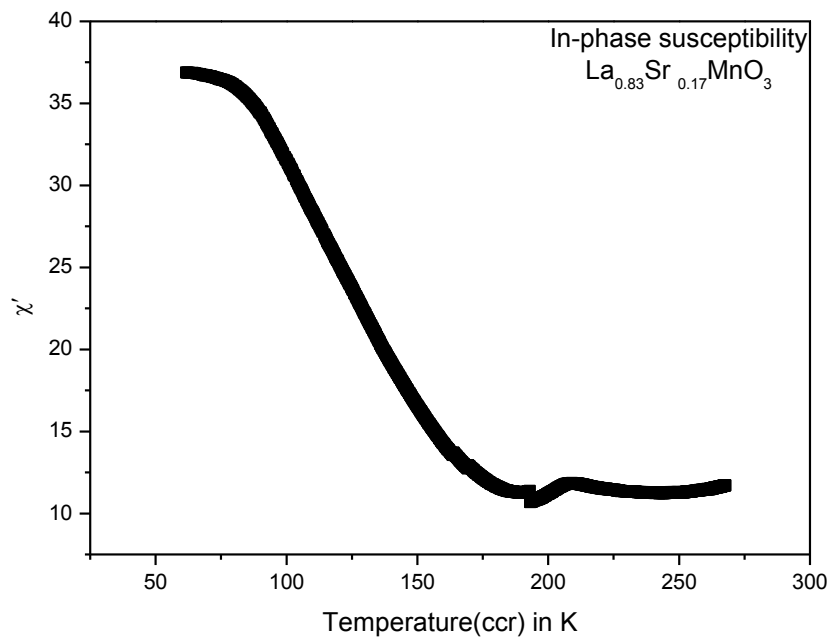


Fig 4.3 Shows the In-phase component of ac susceptibility for $\text{La}_{0.83}\text{Sr}_{0.17}\text{MnO}_3$ sample.

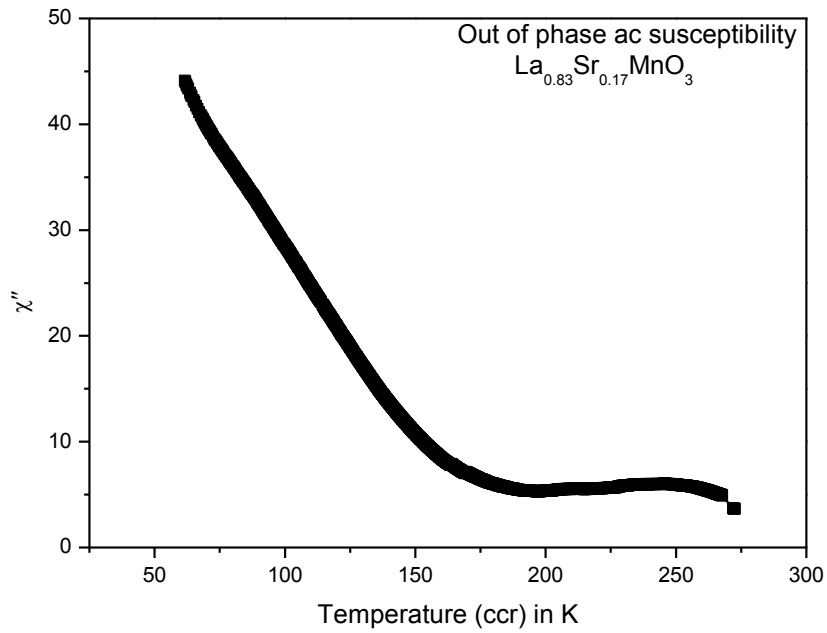


Fig 4.4 Shows the Out of phase component of ac susceptibility for $\text{La}_{0.83}\text{Sr}_{0.17}\text{MnO}_3$ sample.

Around 180 K, the susceptibility starts rising sharply and till the lowest temperature of measurement no saturation is observed. The rise is similar both for in-phase and out-of-phase components of susceptibility. This reflects ferromagnetic nature of LSMO, as per literature [9]. We get the transition temperature of the $\text{La}_{0.83}\text{Sr}_{0.17}\text{MnO}_3$ sample around 183K, but according to the literature, transition temperature is 260K. This variation is due to the temperature gradient between the Cryocooler base (where the temperature sensor is placed) and the sample, which is placed 20 centimeter above the base, inside a poor conducting Hylum.

4.2 CONCLUSION:

A cryocooler based ac susceptometer has been successfully designed and fabricated. We made two designs. In the 1st design the system is brought to low temperature by dipping it in liquid nitrogen. For designing a cryocooler based ac susceptometer 2nd set up is designed in the cryocooler. The former material chosen is hylum because of its high thermal conductivity and low electrical conductivity which can avoid eddy current. After the design, copper winding (150 micron) is done over primary and secondary coils. 1200 turns of copper wire is wound over primary. The secondary coil is a two coil system and the coiling area is separated by some distance 5mm. It had 500 turns wound in clockwise direction and another 500 turns in counter-clockwise direction so that the net magnetization due to the secondary is nullified.

LSMO sample is prepared by auto combustion sol-gel method. Pellets are made, which is then cut into rectangular pieces and is placed in sample holder. One of these pieces is inserted in the secondary coil and AC susceptibility measurement is done.

Susceptibility verses temperature is plotted which is recorded by the cryocooler. A transition is shown around 180 K for $\text{La}_{0.83}\text{Sr}_{0.17}\text{MnO}_3$ sample, slightly deviating the literature value because of the temperature gradient between the cryocooler and the sample which is discussed above.

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